§23. Study of Microwave Effect on the Synthesis of Nanoparticles

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Recent years have witnessed active research in the synthesis of nanoparticles using the effective heating provided by microwave radiation. Parameters that affect the size and size distribution of the dispersed silver nanoparticles that are formed have been described. In all earlier studies the role of the microwave radiation, other than as a heat source, was only of minor interest in organic syntheses and in the syntheses of nanoparticles.

Our overall strategy in this study was to clarify some of the characteristics of the microwave methodology in nanoparticle synthesis.[1)] Accordingly, the principal objective was to use the synthesis of silver nanoparticles in the presence of CMC, which acts as both the reducing agent and a stabilizer of the colloids, as the model reaction with which to examine the thermal features of the microwaves in such a process. In particular, the thermal features of the 2.45-GHz microwave radiation (MW) used in the synthesis of silver nanoparticles are compared to conventional heating (CH), with a special emphasis on the temperature effects and the characteristics (if any) of different microwave synthesizers.

The effect(s) of the 2.45-GHz microwave (MW) radiation in the synthesis of silver nanoparticles in aqueous media by reduction of the diaminesilver(I) complex, $[Ag(NH_3)_2]^+$, with carboxymethylcellulose (CMC) in both batch-type reactor systems with a particular emphasis on the characteristics of the microwaves in this process and the size distributions. The effect of the microwave electric and magnetic fields in the silver nanoparticles were examined with single-mode resonator.

High pure grade $AgNO_3$ (3.14 g), aqueous NH_3 (28%; 3 mL), and ultrapure H_2O (10 mL) were used to prepare the diaminesilver (I) complex, following which a 40-mL solution (60 mM) of $[Ag(NH_3)_2]^+$ was added to aqueous carboxymethylcellulose (CMC; 0.05% w/v; volume of CMC solution, 40 mL). Continuous microwave irradiation with semiconductor generator was irradiated to the sample solution into the quartz reflux reactor. The size distributions and morphologies of the silver nanoparticles were characterized by transmission electron microscopy (TEM). Particle size distributions were also determined by light scattering techniques. Further confirmation of the formation of the Ag nanoparticles was provided by an analysis of the localized surface plasmon resonance (LSPR) absorption recorded on an Agilent 8453 UV-visible spectrophotometer.

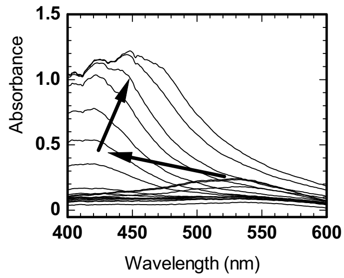

Figure 1 – Temporal change of UV-visible absorption spectra of the silver nanoparticles produced by microwave heating

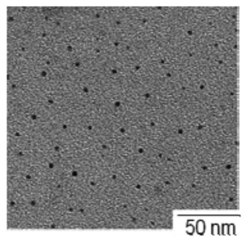

Figure 2 – TEM image of Ag nanoparticles by the MW method

The Plasmon absorption of Ag nano-particles in the microwave electric field is shown in Figure 1. Plasmon absorption at 635nm of Ag seed was observed in the initial stage of the microwave irradiation. When the temperature of a reactive solution did not reached to boiling point (ca. 100° C), Ag nanoparticle was formed by the microwave heating. The formation of the particle below the boiling point is a microwave specific effect. Afterwards, the absorption of 635nm was shifted to 420 nm because the seed Ag particle was grown to Ag nanoparticles. A uniform Ag nano-particle formation was observed in the TEM image.

1) Horikoshi, S. et. Al. : Nanoscale (2010) in press.